

18



Europäisches Patentamt  
European Patent Office  
Office européen des brevets

11 Publication number:

**0 088 556  
B1**

12

## EUROPEAN PATENT SPECIFICATION

45 Date of publication of patent specification: 20.09.89

51 Int. Cl.<sup>4</sup>: A 61 K 9/44

21 Application number: 83300951.7

22 Date of filing: 23.02.83

54 Tablets having clear impressed marks and method for making same.

30 Priority: 08.03.82 JP 37046/82

43 Date of publication of application:  
14.09.83 Bulletin 83/37

45 Publication of the grant of the patent:  
20.09.89 Bulletin 89/38

84 Designated Contracting States:  
BE CH DE FR GB IT LI NL SE

58 References cited:  
EP-A-0 060 023  
FR-A-1 214 530  
US-A-3 125 490  
US-A-4 168 321

73 Proprietor: SUMITOMO CHEMICAL COMPANY,  
LIMITED  
Kitahama 4-chome 5-33  
Chuo-ku Osaka 541 (JP)

72 Inventor: Toya, Kazutoshi  
18-1(1-402), Tamagawa 1-chome  
Takatsuki-shi Osaka 569 (JP)  
Inventor: Uchiyama, Nobuo  
11-8-306, Sonehigashimachi 2-chome  
Toyonaka-shi Osaka 561 (JP)  
Inventor: Miura, Seiko  
18-1(1-112), Tamagawa 1-chome  
Takatsuki-shi Osaka 569 (JP)  
Inventor: Mitsunaga, Takayoshi  
4-23, Yamatedai 6-chome  
Ibaraki-shi Osaka 568 (JP)  
Inventor: Tobiki, Hisao  
4-12-308, Konancho 2-chome  
Higashinada-ku Kobe-shi Hyogo 658 (JP)

74 Representative: Myerscough, Philip Boyd et al  
J.A.Kemp & Co. 14, South Square Gray's Inn  
London, WC1R 5EU (GB)

Note: Within nine months from the publication of the mention of the grant of the European patent, any person may give notice to the European Patent Office of opposition to the European patent granted. Notice of opposition shall be filed in a written reasoned statement. It shall not be deemed to have been filed until the opposition fee has been paid. (Art. 99(1) European patent convention).

Courier Press, Leamington Spa, England.

**EP 0 088 556 B1**

## Description

This invention relates to a method of making tablets having clear marks (including letters and figures) impressed thereon.

5 Generally, tablets are impressed with marks for identification of, for example, kinds and contents of ingredients and names of makers. In some case identification is made by observing marks appearing on the surface of a film coated on tablets which have been impressed with the marks.

Such identification is difficult because marks are shown only by the irregularity of the surface of tablets which is produced by impressing the marks, and when a coating film is provided thereon, the coating cannot be large because the impressed valley portions are filled with the coating agent.

10 Another method of identification is to print marks on the surface of tablets provided with a coating. This method has the problems that the printing ink often falls off due to friction between tablets during handling to result in unclear printed marks and furthermore to cause stains on other tablets, and that the tablets may stick to an off-set printing roll due to low peeling property between the coating on the surface of the tablets and the printing ink.

The invention provides a method for producing tablets having a mark impressed thereon characterised by mixing and contacting tablets having an impressed valley portion providing said mark with a dry powdery material different in color tone from the portion other than the impressed valley portion and then removing excess powdery material not deposited in the impressed valley portion.

20 The material to be deposited in the impressed valley portion has no limitation as long as it is, for example, the one usually used as a coating or is an additive usually used for tablets. This material will be called merely "deposition material" hereinafter. A single deposition material can be used or a mixture of two or more. Usually, there is used an additive to which a coloring matter is added to provide a mark of different color. As examples of dry powdery material, mention may be made of, for example, starches such as corn starch, wheat starch and potato starch, sugars such as lactose, sucrose and mannitol, inorganic coloring matters such as calcium sulfate, calcium phosphate, calcium carbonate, magnesium carbonate, magnesium silicate, magnesium oxide, aluminium hydroxide, titanium oxide, talc, kaolin and bentonite, celluloses such as methyl cellulose, ethyl cellulose, carboxymethylethyl cellulose, hydroxypropylmethyl cellulose, hydroxypropylmethyl cellulose phthalate, hydroxypropyl cellulose and crystalline cellulose, coloring agents such as food dyes and food lake dyestuff, waxes and gum arabic, or mixtures thereof which are *per se* powders or are powdered. Preferred are inorganic coloring matters such as talc, magnesium carbonate, magnesium silicate, magnesium oxide and aluminum hydroxide, hydroxypropyl cellulose or starches. Any other materials which can be deposited in the impressed valley portion may also be used.

35 The tablets impressed with marks (which will be called merely "impressed tablets" hereinafter) have no special limitation in their shape or size, and may be uncoated tablets or coated tablets as long as they have marks impressed thereon. However, adherence of the deposition material to the impressed valley portion of coated tablets is better than that to uncoated tablets. Therefore, in order to obtain clearer impressed marks, it is preferred to use tablets provided with a coating in such a manner that the impressed valley portion is not filled up with the coating.

40 The tablets of this invention are obtained by depositing the deposition material in the impressed valley portion. For this purpose, impressed tablets and the deposition material are mixed and contacted with each other at substantially dry state in a suitable vessel. One method for the mixing and contacting the tablets with the deposition material comprises introducing the impressed tablets and the deposition material in the generally used coating pan or through-flow drying type pan and operating the pan until the deposition material has been uniformly deposited in the impressed valley portion.

45 The amount of the deposition material relative to the tablets at mixing and contacting varies depending on the surface properties of the tablets and on the deposition material and usually is 5% or less. Of course, addition of more than 5% causes no problems.

50 Then excess deposition material (other than deposited in the impressed valley portion) is removed. This may be carried out, for example, by sifting excess deposition material from the tablets with an ordinary sieve, by rubbing the surface of the tablets with a brush and by passing air therethrough, for example by inserting into a layer of tablets in a coating pan an exhaust tube the open tip of which is covered with a gauze or a net and removing the excess deposition material by suction. More preferably, excess deposition material is easily removed by simultaneous charging and discharging of air using a through-flow type pan or apparatus.

55 The tablets having an impressed valley portion in which a deposition material is deposited by the method of the invention have, as they are, sufficiently clear impressed marks, but if desired they may be provided with a desired coating such as a water-soluble, gastric juice soluble or enteric coating. Amount of the coating has no special limitation as long as it does not cause loss of the difference in color tone which brings about the clear impressed marks. Any material generally used for coating tablets may be used as components of the coating. As examples of coating agents, mention may be made of sucrose, methyl cellulose, hydroxypropylmethyl cellulose, hydroxypropyl cellulose, polyvinyl acetal diethylaminoacetate, carboxymethylethyl cellulose, cellulose acetate phthalate, hydroxypropylmethyl cellulose phthalate, methacrylic acid and ethyl acrylate copolymers. As examples of plasticizers, mention may be made of polyethylene glycol, propylene glycol, glycerine, triacetin, castor oil, myvacet and shellac, and as examples

## EP 0 088 556 B1

of coloring agents, mention may be made of food dyestuff, food lake dyestuff, titanium oxide, talc and kaolin. Any coating solvents which are generally used such as water, ethanol, acetone, methylene chloride and isopropyl alcohol may be used. As coating method, there may be used any methods such as pouring of coating solution, spraying by air spray or airless spray gun. Any of coating devices recently used for  
5 coating such as through-flow type coating pans or fluidization type coating devices may be used besides the conventionally used so-called coating pans. The coating conditions are the same as those conventionally employed.

When a mixture of a wax and a powdery material or powder is used as the deposition material in this invention, adherence of the deposition material and the impressed tablets in the impressed valley portion  
10 can be further increased by heating the wax mixture after deposition in the valley portion to 40—90°C to thereby melt the wax. Preferred example of this method comprises depositing the wax mixture in the impressed portion and then removing an excess deposition material other than deposited in the valley portion by the above mentioned method before heating it to 40—90°C. Thus obtained impressed tablets may be coated, if necessary. The heating to 40—90°C may be carried out by the ordinary drying method for  
15 tablets such as by passing warm air therethrough or by leaving them in a warming chamber. The waxes used for this purpose are those which are solid at room temperature and have a melting point of 90°C or less. Examples of the waxes are oils and fats such as hydrogenated oil (Lubri Wax®), etc., bees wax and Carnuba wax, hydrocarbons such as paraffins, higher alcohols such as cetyl alcohol and stearyl alcohol, higher fatty acids such as stearic acid, and palmitic acid, polyhydric alcohols such as polyethyleneglycol,  
20 fatty acid esters of polyhydric alcohols, e.g. sucrose fatty acid esters such as sucrose monopalmitate, sucrose monostearate, sucrose tripalmitate and sucrose tristearate, and sorbitan fatty acid esters such as sorbitan monostearate, sorbitan monopalmitate and sorbitan tristearate.

The waxes may be used in an amount of 70% or less, preferably 5—50% of other powdery material or powder.

25 Preferred examples of said powdery material or powder are inorganic colorants, starches, celluloses or mixtures thereof with colorants. These may be deposited in the same manner as mentioned hereinbefore.

Thus obtained tablets have clearer marks for identification than the conventional tablets and may be subjected to polishing to give gloss.

30 This invention will be illustrated by the following Examples wherein parts are by weight.

### Example 1 (Preparation of tablets having marks impressed thereon)

35	Lactose	70 parts
	Corn starch	25 parts
	Carboxymethyl cellulose calcium	5 parts

The above components were mixed and to the mixture was added 20 parts of 5% corn starch paste. The mixture was kneaded and then dried to obtain granules, to which 0.5 part of magnesium stearate was  
40 added followed by mixing. From the mixture were prepared tablets having a diameter of 8 mm and a weight (per tablet) of 190 mg and impressed with a figure "50" (width: 0.3 mm, depth: 0.15 mm and angle: 60°) on the surface by a rotary tablet machine.

### (Preparation of coating solution)

45	Polyvinyl acetal diethylaminoacetate	6 parts
	Polyethylene glycol 6000	1 part
	Titanium oxide	0.2 part
50	Methanol	93 parts

The above components were stirred until they were homogeneously dispersed or dissolved to prepare a coating solution.

### (Procedure)

55 1.5 kg of the tablets and 70 g of a deposition material which was a mixture of 100 parts of talc and one part of FD & C yellow No. 5 aluminum lake dyestuff were charged in a coating pan of about 30 cm in diameter and the coating pan was operated for 10 minutes to uniformly deposit said deposition material in the valley portion of the impressed figure. Then, the tablets were taken out from the coating pan and excess deposition material was removed by sifting by a No. 12 sieve. Then, the tablets were again charged in the  
60 coating pan to carry out coating with said coating solution according to a conventional method to obtain tablets having a coating amount of about 4.1 mg per one tablet and having the impressed figure colored in reddish yellow when 600 g of said coating solution had been sprayed.

65

## EP 0 088 556 B1

### Example 2 (Preparation of coating solution)

5	Hydroxypropylmethyl cellulose	7 parts
	Titanium oxide	1 part
	Water	92 parts

The above components were stirred until they were homogeneously dispersed or dissolved to obtain a coating solution.

#### (Procedure)

10 kg of the same tablets as used in Example 1 were charged in High-Coater® (through-flow drying type coating device, HCT-60, manufactured by Freund Sangyo K.K.) and coating was effected according to the conventional method to obtain tablets having a coating amount of about 5.1 mg per one tablet when 5 kg of said coating solution was sprayed. Then, after discontinuation of charging and discharging of air, to the tablets was added 200 g of a deposition material prepared by mixing 100 parts of kaolin, 3 parts of FD & C blue No. 2 aluminum lake dyestuff and 10 parts of corn starch and the pan was operated for 5 minutes to uniformly deposit the deposition material in the impressed valley figure portion. Then, excess deposition material was removed by operating the pan for 10 minutes with passing air therethrough by charging and discharging of air to obtain tablets having the impressed figure colored in blue.

### Example 3 (Preparation of coating solution)

25	Hydroxypropylmethyl cellulose phthalate	5 parts
	Titanium oxide	0.3 part
	Methylene chloride	45 parts
	Ethanol	45 parts

The above components were stirred until they were homogeneously dispersed or dissolved to obtain a coating solution.

#### (Procedure)

10 kg of the same tablets as used in Example 1 were charged in High-Coater® (HCT-60) to coat them with the same coating solution as used in Example 2 according to a conventional method to obtain tablets having a coating amount of about 2.2 mg per one tablet when 2 kg of the coating solution was sprayed. Then, after discontinuation of charging and discharging of air, to the tablets was added 200 g of a deposition material prepared by mixing 100 parts of kaolin, 3 parts of FD & C yellow No. 5 aluminum lake dyestuff and 20 parts of lactose and the pan was operated for 5 minutes to uniformly deposit the deposition material in the impressed valley figure portion. Thereafter, the pan was further operated for 10 minutes with passing air therethrough by charging and discharging of air to remove excess deposition material. Then, the tablets were coated with 32 kg of said coating solution prepared in this Example according to a conventional method to obtain tablets having a coating amount of about 23.0 mg per one tablet and having the impressed figure colored in reddish yellow. These tablets met the test specification for enteric coated preparations disclosed in the Japanese Pharmacopoeia.

### Example 4 (Preparation of coating solution)

50	Coating solution—1	
	Hydroxypropylmethyl cellulose	6 parts
	Titanium oxide	0.3 part
	Red No. 103 dyestuff (The Japanese Standard of Food Additives)	1.5 parts
55	Polyethylene glycol 400	1.5 parts
	Water	90 parts
	Coating solution—2	
60	Hydroxypropylmethyl cellulose	7 parts
	Water	93 parts

The above components were stirred until they were homogeneously dispersed or dissolved to obtain the coating solution—1 and the coating solution—2.

## EP 0 088 556 B1

### (Procedure)

15 kg of the same tablets as used in Example 1 were charged in High-Coater® (HCT-60) to coat them with coating solution—1 by the conventional method to obtain tablets having a coating amount of about 2.1 mg per one tablet when 3 kg of the coating solution—1 was sprayed. Then, after discontinuation of charging and discharging of air, 300 g of talc was added to the tablets and the pan was operated for 5 minutes to uniformly deposit the talc in the impressed valley figure portion. Thereafter, the pan was further operated for 2 minutes with passing air therethrough by charging and discharging of air to remove excess talc. Then, coating of the tablets was carried out using 1 kg of said coating solution—2 to obtain reddish brown tablets having a coating amount of about 2.7 mg per one tablet and having the impressed figure colored in white.

The above procedure was repeated except that talc was replaced with 300 g of magnesium silicate, magnesium oxide or aluminum hydroxide to obtain reddish brown tablets having white impressed figure in each case.

### Example 5 (Preparation of coating solution)

Hydroxypropyl cellulose	8 parts
FD & C yellow No. 5 aluminum lake dyestuff	1 part
Glycerine	0.5 part
Water	90 parts

The above components were stirred until they were homogeneously dispersed or dissolved to obtain a coating solution.

### (Procedure)

10 kg of the same tablets as used in Example 1 were charged in High-Coater® (HCT-60) to coat them with said coating solution by a conventional method to obtain tablets having a coating amount of about 4.2 mg per one tablet when 4 kg of the coating solution was sprayed. Then, after discontinuation of charging and discharging of air, to the tablets was added 300 g of a deposition material obtained by mixing 100 parts of talc and 4 parts of FD & C blue No. 1 aluminum lake dyestuff and the pan was operated for 5 minutes to uniformly deposit the material in the impressed valley figure portion. Then, the pan was further operated for 10 minutes with passing air by charging and discharging of air to obtain reddish yellow tablets having blue impressed figure portion.

### Example 6 (Preparation of tablets having impressed mark)

Lactose	70 parts
Corn starch	30 parts
FD & C blue No. 1 aluminum lake dyestuff	0.5 part

The above components were mixed and to the mixture was added 20 parts of 5% corn starch paste. They were kneaded and then dried to obtain granules. 0.5 part of magnesium stearate was added thereto and mixed. From the mixture, blue tablets having a diameter of 8 mm and a weight (per one tablet) of 200 mg and having an impressed figure "246" (width: 0.32 mm, depth: 0.16 mm and angle: 60°) on their surface were produced by a rotary tablet machine.

### (Preparation of coating solution)

Methyl cellulose	4 parts
Water	96 parts

The above components were stirred until they were dissolved to obtain a coating solution.

### (Procedure)

4 kg of said tablets and 170 g of heavy magnesium carbonate were charged in a coating pan of about 40 cm in diameter and the coating pan was operated for 10 minutes to uniformly deposit the heavy magnesium carbonate in the impressed valley figure portion. Then, an exhaust tube the opening end of which was covered with a gauze was inserted in the tablets to remove excess heavy magnesium carbonate by suction air. Thereafter, the tablets were coated by spraying 600 g of said coating solution thereon by a conventional method to obtain blue tablets having white impressed figure portion.

## EP 0 088 556 B1

### Example 7 (Preparation of coating solution)

5	Hydroxypropylmethyl cellulose	6 parts
	Titanium oxide	0.2 part
	Yellow iron oxide	1.5 parts
	Polyethylene glycol 6000	3 parts
	Water	90 parts

10 The above components were stirred until they were homogeneously dispersed or dissolved to obtain a coating solution.

#### (Procedure)

15 From the granules prepared in Example 1, tablets having a diameter of 8.5 mm and a weight (per one tablet) of 210 mg and having an impressed bisect line (width: 0.5 mm, depth: 0.25 mm and angle: 90°) on their surface were produced by a rotary tablet machine. 10 kg of these tablets were charged in High-Coater® (HCT-60) to coat them with said coating solution by a conventional method to obtain tablets having a coating amount of about 5 mg when 3 kg of the coating solution was sprayed. Then, after discontinuation of charging and discharging of air, 400 g of calcium carbonate was added to the tablets and the pan was  
20 operated for 10 minutes to uniformly deposit calcium carbonate in the impressed valley line portion. Then, the pan was further operated for 2 minutes with passing air therethrough by charging and discharging of air to remove excess calcium carbonate. Then, these tablets were coated with 1 kg of the coating solution used in Example 6 by a conventional method to obtain yellowish brown tablets having a coating amount of about 6 mg per one tablet and having a white impressed line portion.

### Example 8 (Preparation of coating solution)

30	Coating solution—1	
	Hydroxypropylmethyl cellulose	7 parts
	Iron sesquioxide	2 parts
	Water	91 parts
35	Coating solution—2	
	Eudragit L30D55® (aqueous dispersion of methacrylic acid and ethyl acrylate copolymer manufactured by Röhm Pharma GMBH)	50 parts
	Water	50 parts

40 The above components were stirred until they were homogeneously dispersed and dissolved to obtain coating solutions—1 and —2.

#### (Procedure)

45 From the granules prepared in Example 6, tablets having a diameter of 9 mm and a weight (per one tablet) of 280 mg and having an impressed figure "510" (width: 0.2 mm, depth: 0.1 mm and angle: 50°) on their surface were prepared by a rotary tablet machine. 12 kg of these tablets were charged in High-Coater® (HCT-60) to coat them with said coating solution—1 by a conventional method to obtain tablets having a coating amount of about 4 mg per one tablet when 3 kg of the coating solution was sprayed. Then, after discontinuation of charging and discharging of air, 600 g of lactose was added to the tablets and the pan was  
50 operated for 2 minutes to uniformly deposit the lactose in the impressed valley figure portion. Thereafter, the excess lactose was removed with passing air therethrough by charging and discharging of air. Then, these tablets were coated with 7 kg of said coating solution—2 by a conventional method to obtain reddish brown tablets having a coating amount of about 2.1 mg per one tablet and having a white figure portion.

55 The above procedure was repeated using 600 g of hydroxypropyl cellulose (L-HPC® manufactured by Shinetsu Chemical Co., Ltd.) in place of the lactose to obtain reddish brown tablets having a white figure portion.

60 These tablets met the test specification for enteric coated preparations mentioned in Japanese Pharmacopoeia.

65

## EP 0 088 556 B1

### Example 9 (Preparation of coating solution)

5	Hydroxypropyl cellulose	5 parts
	Stearic acid	0.5 part
	Ethyl alcohol	40 parts
	Methylene chloride	60 parts

The above components were stirred until they were homogeneously dissolved to obtain a coating solution.

#### (Procedure)

From the granules prepared in Example 1, tablets having a diameter of 10 mm and a weight (per one tablet) of 360 mg and having an impressed figure "135" (width: 0.43 mm, depth: 0.23 mm and angle: 60°) on their surface were prepared by a rotary tablet machine. 15 kg of these tablets were charged in High-Coater® (HCT-60) to coat them with said coating solution by a conventional method to obtain tablets having a coating amount of about 2 mg per one tablet when 2.5 kg of the coating solution was sprayed. After discontinuation of charging and discharging of air, to these tablets was added 500 g of a deposition material prepared by adding about 10 parts of water to 100 parts of mannitol and 5 parts of FD & C yellow No. 5 dyestuff, mixing and drying them and grounding them and the pan was operated for 2 minutes to uniformly deposit the material in the impressed valley figure portion. Then, the pan was further operated for one minute with passing air therethrough by charging and discharging of air to remove excess material. Thereafter, these tablets were further coated with 1 kg of said coating solution by a conventional method to obtain tablets having a coating amount of about 3 mg per one tablet and having reddish yellow impressed figure portion.

### Example 10 (Preparation of coating solution)

30	Hydroxypropylmethyl cellulose	5 parts
	Iron sesquioxide	1 part
	Talc	0.5 part
	Water	90 parts

The above components were stirred until they were homogeneously dispersed or dissolved to obtain a coating solution.

#### (Procedure)

From the granules prepared in Example 1, tablets having a diameter of 8 mm and a weight (per one tablet) of 200 mg and impressed with a figure "124" (width: 0.36 mm, depth: 0.18 mm and angle: 60°) on the surface were prepared by a rotary tablet machine. 12 kg of these tablets were charged in High-Coater® (HCT-60) to coat them with said coating solution by a conventional method to obtain tablets having a coating amount of about 1 mg per one tablet when 1.5 kg of said coating solution was sprayed. After discontinuation of charging and discharging of air, to the tablets was added 200 g of a mixture of 10 parts of talc and 1 part of a sucrose fatty acid ester (DKF-50®-manufactured by Daiichi Kogyo Seiyaku Co., Ltd.) and the pan was operated for 3 minutes to uniformly deposit the mixture in the impressed valley figure portion. Thereafter, the pan was further operated for 1 minute with passing air therethrough by charging and discharging of air to remove excess mixture followed by passing therethrough hot air of 90°C for 15 minutes to obtain reddish brown tablets having a white impressed figure portion.

The above procedure was repeated using a hydrogenated oil (Lubri Wax 101® manufactured by Freund Sangyo K.K.) in place of the sucrose fatty acid ester to obtain reddish brown tablets having a white impressed figure portion.

### Example 11 (Preparation of coating solution)

55	Hydroxypropylmethyl cellulose	6 parts
	Yellow iron oxide	1.5 parts
	Glycerin	0.5 part
60	Water	90 parts

The above components were stirred until a homogeneous dispersion or solution was obtained to prepare a coating solution.

65

## EP 0 088 556 B1

### (Procedure)

15 kg of the same tablets as used in Example 1 were charged in High-Coater® (HCT-60) to coat them with said coating solution by a conventional method to obtain tablets having a coating amount of about 2 mg per one tablet when 2.5 kg of the coating solution was sprayed. After discontinuation of charging and discharging of air, to the tablets was added 600 g of a ground mixture of 5 parts of magnesium carbonate and 1 part of polyethylene glycol 6000 and the pan was operated for 2 minutes to uniformly deposit said mixture in the impressed valley figure portion. Thereafter, the pan was further operated for 1 minute with passing air therethrough by charging and discharging of air to remove excess mixture followed by passing hot air of 80°C for 15 minutes to obtain yellowish brown tablets having a white impressed figure portion.

### Example 12

#### (Preparation of coating solution)

	Hydroxypropyl cellulose	7 parts
15	FD & C yellow No. 5 aluminum lake dyestuff	0.5 part
	Glycerin	0.5 part
	Water	90 parts

The above components were stirred until they were homogeneously dispersed or dissolved to prepare a coating solution.

### (Procedure)

From the granules prepared in Example 1, tablets having a diameter of 9 mm and a weight (per one tablet) of 280 mg and impressed with a figure "510" (width: 0.2 mm, depth: 0.1 mm and angle: 50°) on the surface were prepared by a rotary tablet machine. 4 Kg of these tablets were charged in a coating pan of about 40 cm in diameter and were coated with 1.2 kg of said coating solution by a conventional method. Then, 180 g of mixture of 1 part of stearic acid and 1 part of magnesium oxide was added to the tablets and the pan was operated for 5 minutes to uniformly deposit the mixture in the impressed valley figure portion. Thereafter, an exhaust tube the opening end of which was covered with a gauze was inserted in the tablets to remove excess mixture by suction air. The resulting tablets were charged in a chamber dryer and heated at 80°C for 5 hours to obtain reddish yellow tablets having a white impressed figure portion.

### Claims

1. A method for producing tablets having a mark impressed thereon characterised by mixing and contacting tablets having an impressed valley portion providing said mark with a dry powdery material different in color tone from the portion other than the impressed valley portion and then removing excess powdery material not deposited in the impressed valley portion.
2. A method according to claim 1 wherein the tablets are provided with a coating after said material has been deposited in the impressed valley portion.
3. A method according to claim 1 wherein the tablets are provided with a coating before deposition of said material in the impressed valley portion.
4. A method according to claim 1, 2 or 3 wherein excess material is removed by passing air through a tablet layer.
5. A method according to any one of the preceding claims wherein the material is deposited using a pan.
6. A method according to claim 5 wherein the pan is a through-flow type pan.
7. A method according to claim 6 wherein excess material is removed by simultaneous charging and discharging of air into and from a through-flow drying type pan.
8. A method according to any one of the preceding claims wherein the powdery material is an inorganic colorant, hydroxypropyl cellulose or starch.
9. A method according to claim 8 wherein the inorganic colorant is talc, magnesium carbonate, magnesium silicate, magnesium oxide or aluminum hydroxide.
10. A method according to claim 8 wherein the powdery material comprises a mixture of a wax which is solid at room temperature and has a melting point of 90°C or less with another material.
11. A method according to claim 10 wherein the mixture contains 5 to 50% by weight of wax, based on the weight of said another material.
12. A method according to claim 10 or 11 wherein the mixture is heated to 40—90°C after its deposition in the impressed valley portion.

### Patentansprüche

1. Ein Verfahren zur Herstellung von Tabletten, die mit einer eingepreßten Markierung versehen sind, dadurch gekennzeichnet, daß die mit einer eingepreßten, die Markierung ergebenden Vertiefung versehenen Tabletten mit einem trockenen Pulvermaterial zusammengebracht und vermischt werden, das



## EP 0 088 556 B1

sich in der Färbung von der Farbe des Tablettenabschnittes mit der eingepreßten Vertiefung unterscheidet, und daraufhin überschüssiges, nicht in der eingepreßten Vertiefung angelagertes Pulvermaterial entfernt wird.

2. Das Verfahren nach Anspruch 1, wobei die Tabletten mit einem Überzug versehen werden, nachdem das Material in der eingepreßten Vertiefung angelagert worden ist.

3. Das Verfahren nach Anspruch 1, wobei die Tabletten mit einem Überzug versehen werden, bevor das Material in der eingepreßten Vertiefung angelagert wird.

4. Das Verfahren nach einem der Ansprüche 1 bis 3, wobei überschüssiges Pulvermaterial entfernt wird, indem Luft durch eine Tablettenschicht geblasen wird.

5. Das Verfahren nach einem der Ansprüche 1 bis 4, wobei die Anlagerung des Pulvermaterials in einer Pfanne erfolgt.

6. Das Verfahren nach Anspruch 5, wobei eine Pfanne verwendet wird, durch welche zwangsweise ein Gas geführt werden kann (through-flow type pan).

7. Das Verfahren nach Anspruch 6, wobei zur Entfernung von überflüssigem Material gleichzeitig Luft in die durchströmbare Pfanne eingeführt und Luft aus dieser durchströmbaren Pfanne herausgeführt wird.

8. Das Verfahren nach einem der Ansprüche 1 bis 7, wobei als Pulvermaterial ein anorganischer Farbträger, Hydroxylpropyl - Cellulose oder Stärke verwendet wird.

9. Das Verfahren nach Anspruch 8, wobei als anorganischer Farbträger Talk, Magnesiumcarbonat, Magnesiumsilikat, Magnesiumoxid oder Aluminiumhydroxid verwendet wird.

10. Das Verfahren nach Anspruch 8, wobei als Pulvermaterial ein Gemisch dient, das neben einem anderen Material ein Wachs enthält, das bei Raumtemperatur fest ist und das einen Schmelzpunkt von 90°C oder weniger aufweist.

11. Das Verfahren nach Anspruch 10, wobei das Gemisch—bezogen auf das Gewicht des anderen Materials—5 bis 50 Gewichts-% Wachs enthält.

12. Verfahren nach Anspruch 10 oder 11, wobei das Gemisch auf 40 bis 90°C erwärmt wird, nachdem es in der eingepreßten Vertiefung angelagert worden ist.

### Revendications

1. Procédé de production de comprimés sur lesquels est imprimé un marquage, caractérisé en ce qu'il consiste à mélanger et à mettre en contact des comprimés ayant une partie imprimée en creux ménageant ce marquage, avec une matière pulvérulente sèche de nuance de coloration différente de la partie autre que la partie imprimée en creux, puis à éliminer la matière pulvérulente en excès qui ne s'est pas déposée dans la partie imprimée en creux.

2. Procédé suivant la revendication 1, qui consiste à munir les comprimés d'un enrobage après que la matière a été déposée dans la partie imprimée en creux.

3. Procédé suivant la revendication 1, qui consiste à munir les comprimés d'un enrobage avant le dépôt de la matière dans la partie imprimée en creux.

4. Procédé suivant la revendication 1, 2 ou 3, qui consiste à éliminer la matière en excès en faisant passer de l'air dans une couche de comprimés.

5. Procédé suivant l'une quelconque des revendications précédentes, qui consiste à déposer la matière en utilisant un chaudron.

6. Procédé suivant la revendication 5, dans lequel le chaudron est un chaudron de type à passage continu.

7. Procédé suivant la revendication 6, qui consiste à éliminer la matière en excès en envoyant de l'air dans un chaudron de type à séchage à passage continu et en l'évacuant simultanément de ce chaudron.

8. Procédé suivant l'une quelconque des revendications précédentes, dans lequel la matière pulvérulente est un colorant minéral, de l'hydroxypropylcellulose ou de l'amidon.

9. Procédé suivant la revendication 8, dans lequel le colorant minéral est du talc, du carbonate de magnésium, du silicate de magnésium, de l'oxyde de magnésium ou de l'hydroxyde d'aluminium.

10. Procédé suivant la revendication 8, dans lequel la matière pulvérulente comprend un mélange d'une cire qui est solide à la température ambiante et qui a un point de fusion de 90°C ou inférieur à 90°C et d'une autre matière.

11. Procédé suivant la revendication 10, dans lequel le mélange contient de 5 à 50% en poids de cire, par rapport au poids de l'autre matière.

12. Procédé suivant la revendication 10 ou 11, qui consiste à porter le mélange entre 40 et 90°C, après son dépôt dans la partie imprimée en creux.